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EFFECT OF COLD WORK ON THE HIGH TEMPERATURE CREEP PROPERTIES OF DILUTE ALUMINUM ALLOYS

Twenty Ninth Technical Report

By

Robert E. Frenkel, Oleg D. Sherby, and John E. Dorn

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Office of Naval Research
Department of the Navy
Washington 25, D. C.

ATTENTION: Mr. Julius Harwood

Dear Sir:

Attached hereto is the Twenty Ninth Technical Report on Contract N7-onr-295, Task Order II, NR-031-048, entitled "Effect of Cold Work on the High Temperature Creep Properties of Dilute Aluminum Alloys".

The wholehearted cooperation of the Office of Naval Research in making these studies possible is sincerely appreciated.

Respectfully submitted,

John E. Dorn

John E. Dorn
Professor of Physical Metallurgy

JED:dk

EFFECT OF COLD WORK ON THE HIGH TEMPERATURE CREEP PROPERTIES OF DILUTE ALUMINUM ALLOYS

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ABSTRACT

The creep rate of annealed and cold-worked aluminum alloys at elevated temperatures can be represented by the equation $\dot{\varepsilon} = S e^{-\Delta H_c/RT} e^{B\sigma}$ where ΔH_c = activation energy for creep, R = gas constant, T = absolute temperature, σ = applied stress, B = stress parameter and S = structure parameter. The activation energy for creep is unaffected by cold work and S is only slightly increased by the previous cold deformation. The principal effect of cold work on the creep resistance of aluminum appears to arise from a decrease in B by this factor. The results are discussed in terms of a new recovery model for creep.

INTRODUCTION

A number of routine investigations(1-6) have been made in order to determine the effect of prior cold work on the creep properties of metals. Since the basic laws for high temperature creep were not then known, the results of such investigations were not readily interpretable. Recent progress in formulating the laws for high temperature creep(7-12) has now provided a better basis for evaluating the effect of cold work on the creep properties of metals. It is the purpose of this investigation to study the effect of prior cold work on each of the significant parameters that determine the high temperature creep behavior of metals.

MATERIALS

Sheets of the high purity aluminum alloys listed in Table I were used in this investigation. These alloys were homogenized, cold rolled from

TABLE I

Composition, Recrystallization Treatment and Grain Size of Aluminum Solid Solution Alloys Investigated+

Alloying Element	Chemical Composition						Recrystallization Treatment (after 30% cold rolling)	Mean Grain Diameter (mm)
	Atomic Percent	Weight percent of impurities						
		Si	Fe	Cu	Mg	Mn		
Pure Al	(99.987)	0.003	0.003	0.006	0.001	---	860°F 30 mins.	0.25
Mg	1.617	0.003	0.004	0.006	---	---	800°F 10 mins.	0.26
Cu	0.101	0.003	0.003	---	0.0006	0.001	800°F 43 mins.	0.29

+ The authors wish to acknowledge their appreciation to the Aluminum Company of America Research Laboratories for the preparation of these alloys and the determination of their chemical composition.

0.100 in. to 0.070 in. in thickness and then recrystallized to about the same grain size. Their chemical composition, recrystallization treatment and grain size are recorded in Table I. Creep specimens were machined with their tensile axes in the rolling direction.

EFFECT OF COLD WORK ON THE ACTIVATION ENERGY FOR CREEP

Extensive investigations on the creep of annealed metals[7-9,11] have shown that the creep strain, ε, for a constant stress σ_c is related to the time, t, and test temperature, T, in accordance with the functional relationship

$$\varepsilon = f(\theta) \qquad \sigma_c = \text{const.} \tag{1}$$

where $$\theta = t e^{-\Delta H/RT} \tag{2}$$

ΔH_c = activation energy for creep

and R = gas constant.

In general, Equation 1 has been found to be valid for annealed materials[11] over the high temperature range of creep (from about 0.45 of the absolute melting temperature up to the melting temperature) where the rates of crystal recovery are rapid. The activation energy for creep of initially annealed metals was found to be constant, independent of temperature, duration of test, creep strain, stress, grain size and the various subgrain structures developed during creep[7,8,11]. Furthermore, ΔH_c was found to be insensitive to minor alloying additions[7,11] and to small amounts of dispersed intermediate phases[9]. Therefore the activation energies for creep of relatively pure metals approached those for the elements, and were shown to exhibit a normal periodic variation with atomic number[11]. Wherever appropriate data were available[11] the activation energy for creep, ΔH_c, agreed well with the activation energy for self-diffusion, ΔH_D, suggesting that the rate-controlling mechanism for the high temperature creep process is that of self-diffusion.

Although the activation energy for creep was found to be insensitive to the series of subgrain structures that were developed during creep of a previously annealed alloy, ΔH_c might nevertheless be affected by more severe structural changes such as those induced by prior cold work. In order to examine this possibility, each of the alloys listed in Table I was creep tested in three different initial states. As shown in Table II, state A refers simply to the as-annealed condition, and the resulting creep curves for this initial condition are shown by the solid symbols of Fig. 1. These data confirm the earlier observations[7-9] that the activation energy for creep of dilute aluminum alloys is about 36,000 calories per mole.

TABLE II

Initial State of the Alloys Tested in Creep

Creep Temp. °K	A Annealed	B Annealed, Prestrained 15% at 78°K and recovered as follows:					C Annealed, Prestrained 15% at 78°K and recovered as follows:				
		T_1 °K	t_1 Hrs.	T_2 °K	t_2 Hrs.	θ_R	T_1 °K	t_1 Hrs.	T_2 °K	t_2 Hrs.	θ_R
422							477	1.98	422	2.00	$8.386 \cdot 10^{-17}$
477		530	1.95	477	2.00	$3.636 \cdot 10^{-15}$	477	2.00	—	—	$8.386 \cdot 10^{-17}$
530		530	2.00	—	—	$3.636 \cdot 10^{-15}$					

Initial states B and C were obtained by prestraining the annealed alloys 15% at 78°K followed by an appropriate recovery treatment for each of the two states as shown in Table II. Such consistent recovery treatments

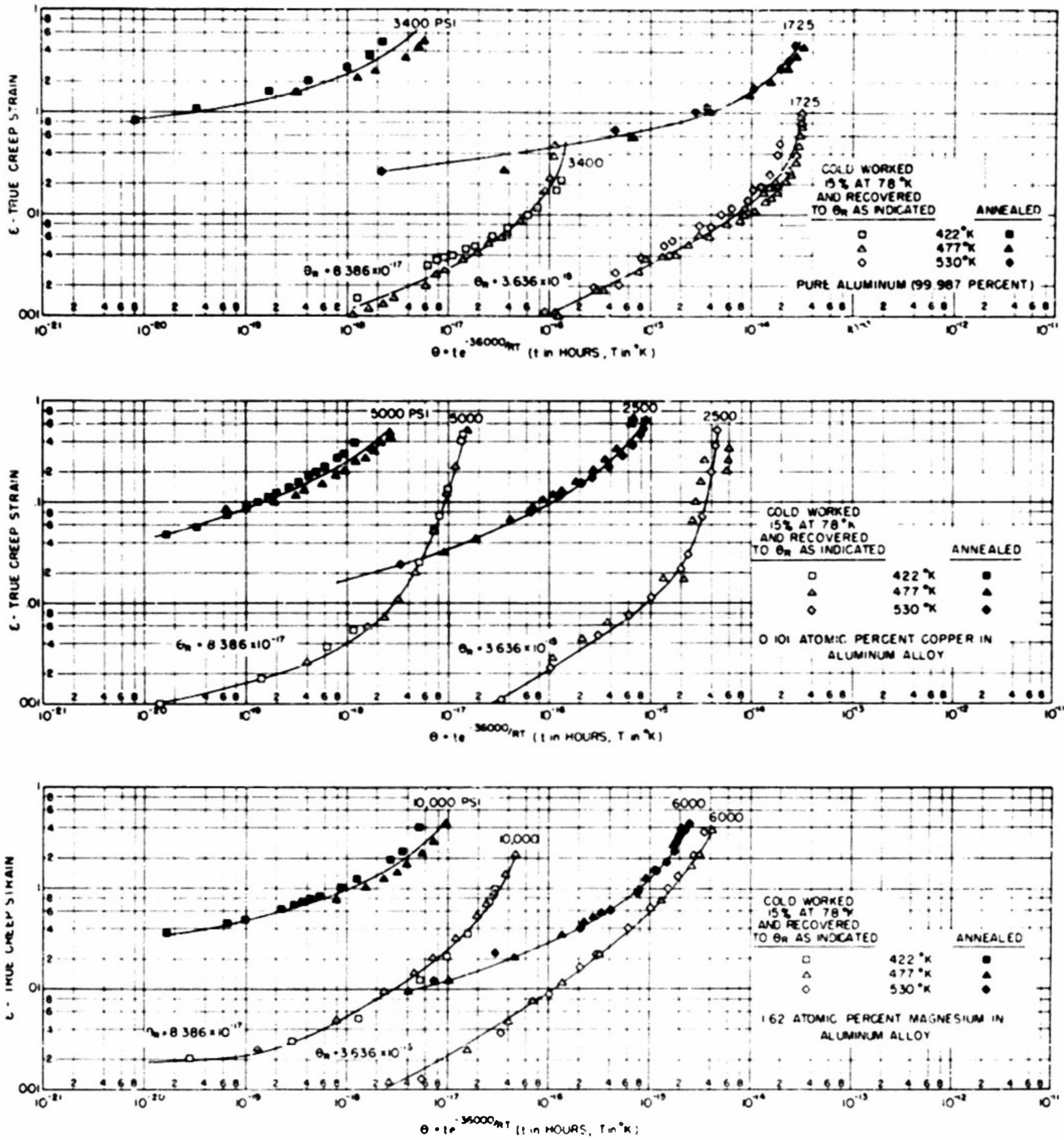

FIG 1 CORRELATION OF CREEP STRAIN - TIME DATA FOR ANNEALED AND COLD-WORKED STATES OF DILUTE ALUMINUM ALLOYS BY MEANS OF THE RELATION $\varepsilon = f(\theta, \sigma_c)$

were necessary to provide the same initial state for each alloy at the creep test temperatures investigated. It was reasonable to expect that the amount of recovery depended on $\theta_R = te^{-\Delta H_R/RT}$ where t is the time of recovery at temperature T, and ΔH_R, the activation energy for recovery, is equal to ΔH_c for creep. This concept is verified by the data recorded in Fig. 2 which represent the tensile stress-strain curves for cold-worked high purity aluminum at 298°K following various recovery treatments. Almost identical stress-strain curves were obtained for two different recovery treatments, one after 1750 hours recovery at 530°K and the other after 117.5 hours recovery at 572°K. These data prescribe an activation energy for recovery equal to about 36,000 calories per mole which is identical with that for creep.

In order [illegible] chi[illegible] standard recovered state in a reasonable period of time, it [illegible] [illegible]asionally desirable to recover for time t_1 at temperature T_1 follo[illegible] [illegible]ecovery for time t_2 at temperature T_2. Under these conditions [illegible] [illegible]ent recovered states were presumed to be established at consta[illegible] [illegible]es of θ_R, where

$$\theta_R = t_1 e^{-\Delta H_R/RT_1} + t_2 e^{-\Delta H_R/RT_2} \tag{3}$$

A[illegible] [illegible]n in Table II, state B refers to recovery to $\theta_R = 3.636 \times 10^{-15}$ [illegible]s state C is that obtained following recovery to $\theta_R = 8.386 \times 10^{-17}$. In Fig. 2 the θ concept for recovery of aluminum is again shown to be valid for these multiple recovery treatments wherein the activation energy for high temperature recovery equals the activation energy for creep. Fig. 2 also reveals that the stress-strain curves at 298°K for the two recovered states are yet appreciably greater than that for the annealed state, showing that considerable effects of the previous cold working were retained following recovery. Additional confirmation of the retention of the effects

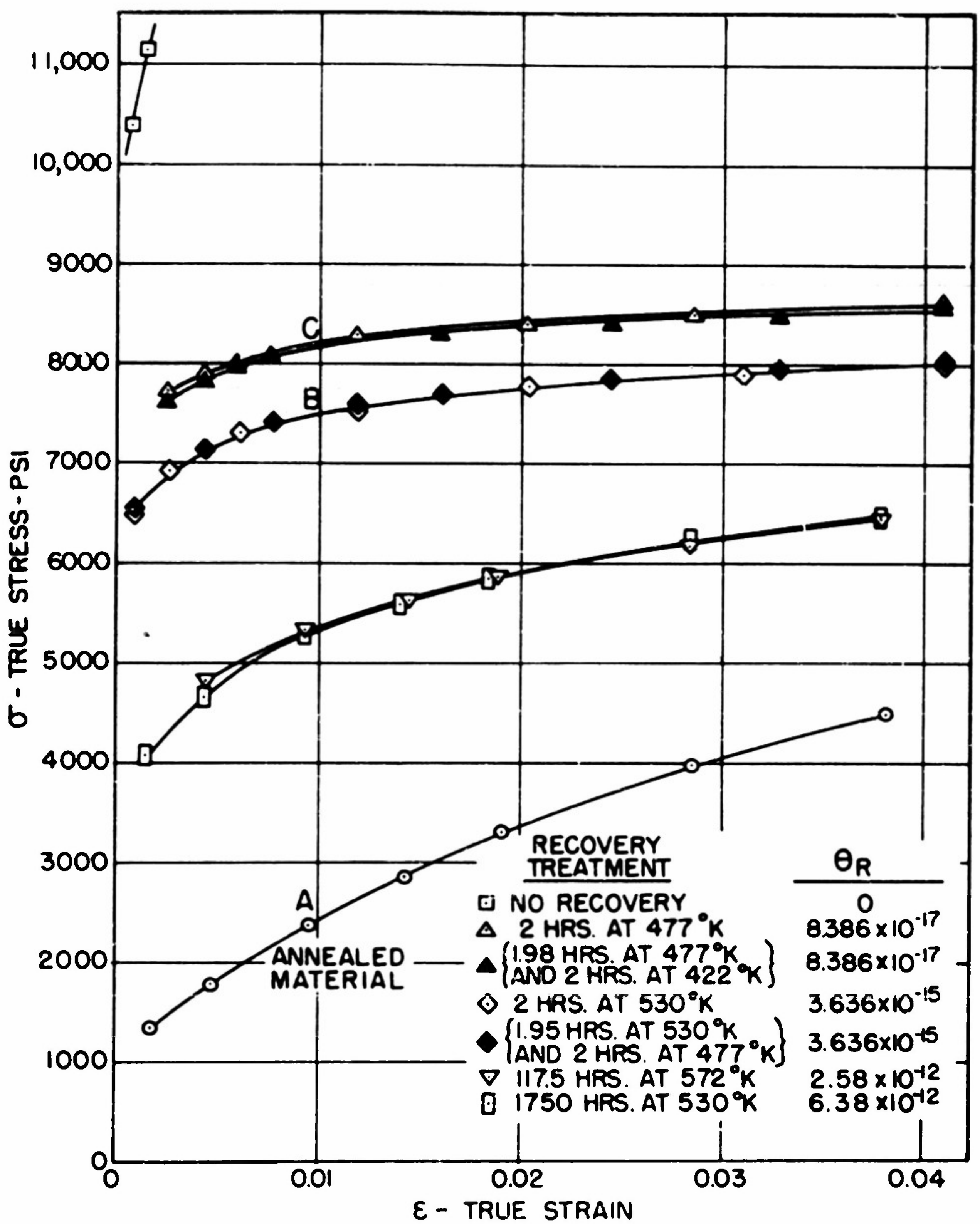

FIG. 2 EFFECT OF RECOVERY ON THE TENSILE CURVES AT 298 °K OF HIGH PURITY ALUMINUM PRESTRAINED 15 % AT 78 °K.

of cold work following recovery to $\theta_R = 8.386 \times 10^{-17}$ is shown by the back-reflection Debye-Scherrer x-ray photograms reproduced in Fig. 3. The annealed coarse-grained structure reveals only relatively few grains so oriented to satisfy Bragg angles. After cold working and recovery broad short diffuse arcs were obtained indicative of the retention of some of the cold working effects.

The creep curves following cold work and recovery are recorded in Fig. 1. Although the creep resistance of each of the alloys was appreciably increased following the cold working and recovery treatments, the activation energy for creep remained about 36,000 calories per mole. Therefore the activation energy for creep is presumed to be a constant independent of the structural changes induced by cold work.

EFFECT OF COLD WORK ON THE STRESS PARAMETER

As shown in Fig. 1 cold work improves the creep resistance, but the activation energy for creep appears to be independent of the structural changes attending cold working. Consequently cold work must affect some other parameter of the creep equation. Previous investigations[10] have demonstrated that the creep rate of initially annealed metals is given by

$$\dot{\varepsilon} = S e^{-\Delta H/RT} e^{B\sigma} \qquad (4)^{*}$$

where B, the stress parameter, was observed to be a constant independent of the instantaneous temperature or the previous creep history of the alloy. Consequently all effects of structural changes attending creep on

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* Equation 4 is valid for values of $B\sigma >> 0$. As $B\sigma$ approaches zero the creep rate must also approach zero. For low stresses therefore, $e^{B\sigma}$ must be replaced by some function that vanishes as $B\sigma$ vanishes such as $\sinh B\sigma$, $(e^{B\sigma} - 1)$ or $B\sigma e^{B\sigma}$.

ANNEALED ALUMINUM

A. BEFORE CREEP TESTING.

B. AT FRACTURE AFTER CREEP UNDER σ_c = 3400 PSI AT 477°K.

ANNEALED ALUMINUM PRESTRAINED 15% AT 78°K AND RECOVERED TO $\theta_R = 8.386 \times 10^{-17}$

A. BEFORE CREEP TESTING.

B. AT FRACTURE AFTER CREEP UNDER σ_c = 3400 PSI AT 477 °K.

FIG. 3 X-RAY BACK REFLECTION PHOTOGRAMS OF ANNEALED AND COLD WORKED ALUMINUM BEFORE AND AFTER CREEP TESTING.

the instantaneous creep rate were found to arise from changes in the parameter S . The reciprocal of B , however, increased practically linearly with atomic percent of small additions of solute elements[10].

Because ΔH_c is unaffected by cold work and yet the creep resistance is improved, either B or S or both of these parameters must be affected by the structural changes attending low temperature straining. In order to study the effect of pretreatment on S it is first necessary to determine whether the structural changes subsequent to cold work and partial recovery affect B . Since the stress parameter B might change with changes in the cold-worked state during creep it was necessary to evaluate B for various creep strains. The procedure adopted in evaluating B during creep of a cold-worked and recovered metal was as follows: Annealed high purity aluminum was prestrained 15% at 78°K, held for two hours at the creep temperature of 477°K and then precrept under $\sigma_c = 4{,}000$ psi to a prescribed strain, following which the stress was reduced and the new instantaneous creep rate was determined. It was assumed that the structural changes attending the precrept condition would remain unaltered immediately following a reduction in stress**. The instantaneous creep rate at the lower stress would thus be characteristic of the particular cold worked, recovered and precrept structure. This procedure was repeated on a new sample for each new value of the reduced stress. The relationship between the instantaneous creep rate and the true stress for the various reduced stresses would therefore pertain to a specific structure. The results for four precrept states of the cold-worked and partially recovered metal are shown in Fig. 4. The linear relationship between the stress

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** This, of course, would not be true upon increasing the stress because the instantaneous increase in strain due to the increase in stress would result in an instantaneous change in the structural state.

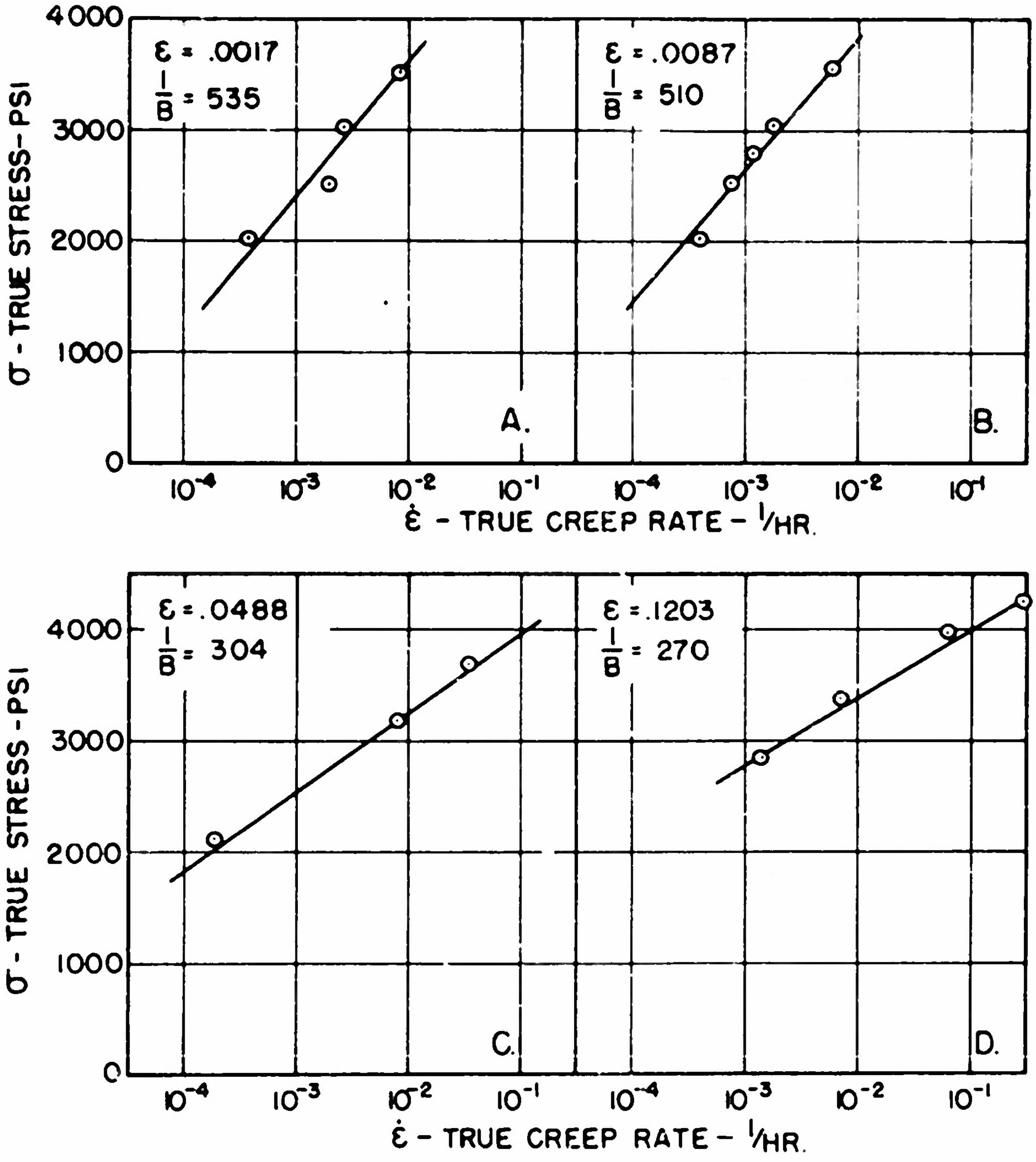

FIG. 4 EFFECT OF TRUE STRESS ON THE TRUE CREEP RATE AT VARIOUS COLD-WORKED STRUCTURES OF ALUMINUM.

(STRUCTURES DEVELOPED BY PRESTRAINING 15% AT 78°K FOLLOWED BY CREEP UNDER σ_C = 4000 PSI AT 477 °K TO CREEP STRAINS INDICATED ABOVE.)

and the logarithm of the strain rate exhibited by the data of Fig. 4 further confirm the earlier validity of the stress term in Equation 4. Cut A of Fig. 4 reveals that $1/B$ is about 535 following a precreep strain of $\varepsilon = 0.0017$ whereas the remaining cuts show that $\frac{1}{B}$ decreases as the precreep strain increases. Since previous tests on annealed high purity aluminum[10] gave $\frac{1}{B}$ equal to about 191 for all creep strains, it follows that cold work results in an increase in $1/B$. As shown in Fig. 5 the value of $1/B$ for the cold-worked aluminum decreases with increasing creep strain and appears to approach the value of $1/B$ for the creep of aluminum in the annealed state. This suggests that the structural modifications induced by cold working are gradually eliminated during high temperature creep. Partial confirmation of this suggestion is contained in the x-ray photograms of Fig. 3, which reveal extensive polygonization for the fractured specimen initially in the annealed state as well as for the ruptured specimen initially cold worked.

EFFECT OF COLD WORK ON THE STRUCTURE PARAMETER

During creep of annealed aluminum ΔH_c and $1/B$ remain constant independent of the creep strain[10]. This fact implies that the parameters ΔH_c and $1/B$ are insensitive to the structural changes that attend the creep of initially annealed aluminum. Consequently the great changes in the creep rate of initially annealed specimens with creep strain are attributable to the effect of structural changes that are induced by creep on S. A typical example of the effect of creep strain for a constant load creep test on S for $\sigma_c = 4{,}000$ psi is shown by curve (b) in Fig. 6. The values of S were calculated by means of Equation 4 using the instantaneous true creep rate, the instantaneous true stress, $\Delta H_c = 36{,}000$ calories per mole, and $1/B = 191$.

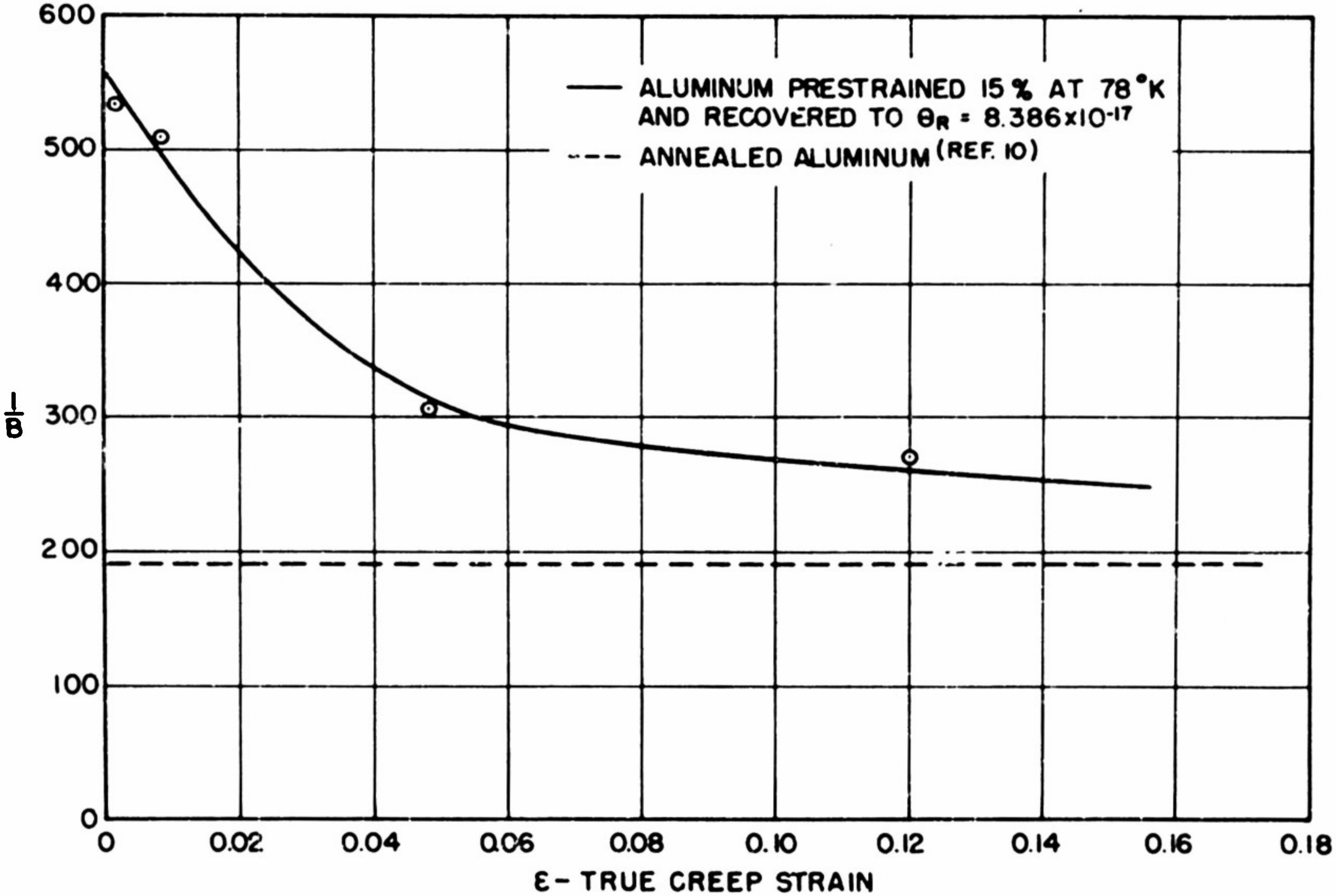

FIG. 5 EFFECT OF CREEP ON $\frac{1}{B}$ FOR COLD-WORKED ALUMINUM UNDER A CREEP STRESS OF σ_C = 4000 PSI AT 477°K.

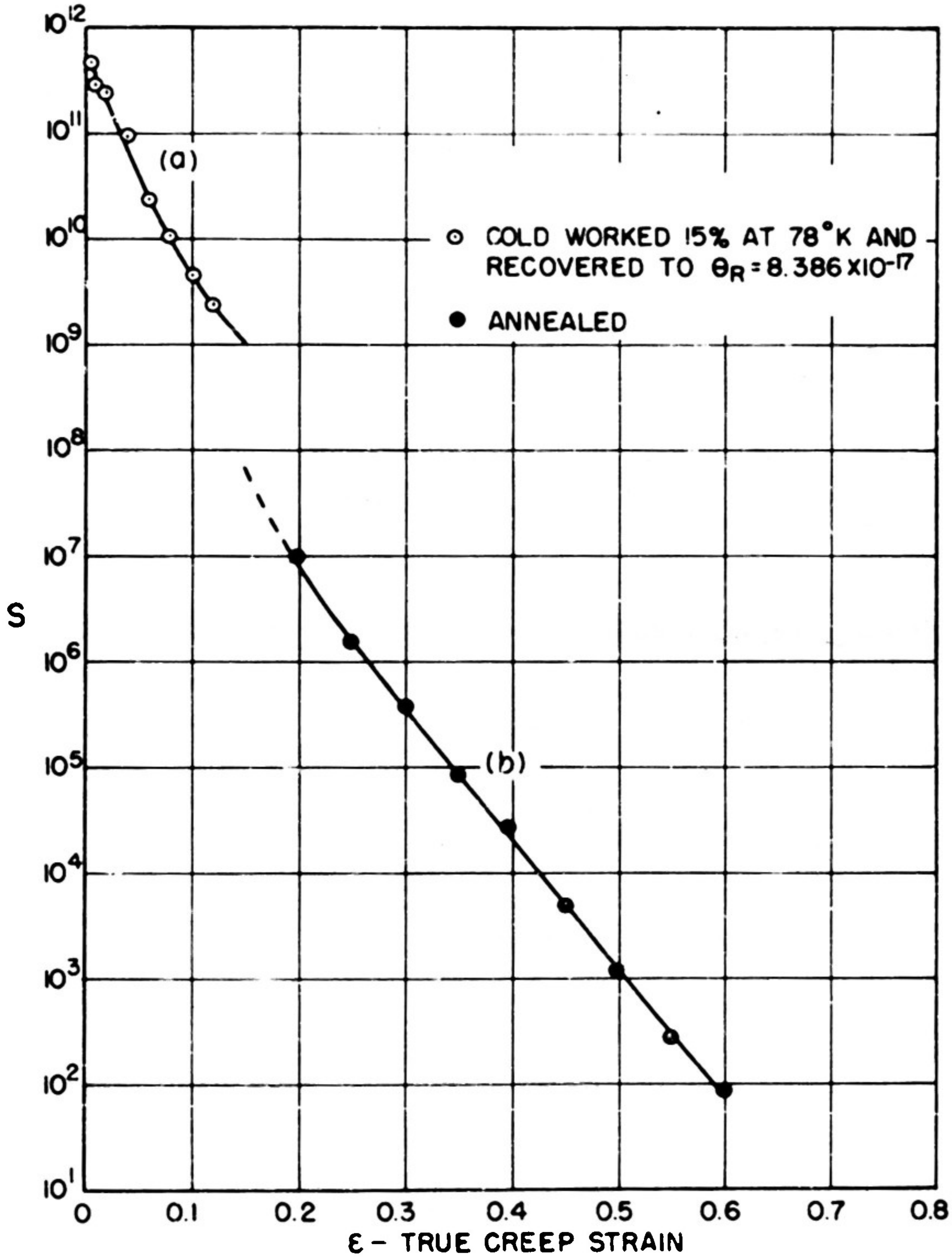

FIG. 6 EFFECT OF CREEP ON S-PARAMETER FOR (a) COLD-WORKED PURE ALUMINUM AND (b) ANNEALED ALUMINUM UNDER A CREEP STRESS $\sigma_C = 4000$ PSI AT 477°K.

The effect of creep strain on S was calculated in an analogous way for the initially cold-worked and partially recovered aluminum under the same constant load condition of $\sigma_c = 4{,}000$ psi. Here however, the instantaneous values of $\frac{1}{B}$ given in Fig. 5 were introduced in Equation 4. As shown in Fig. 6, S for the cold-worked material also appears to decrease with the creep strain. Unfortunately a direct comparison between the annealed and cold-worked aluminum cannot be made because of the high initial strain upon initial loading of the annealed aluminum and the early fracture of the cold-worked aluminum. The extrapolated curves, however, suggest that when comparisons are made at the same creep strain, S appears to be increased merely by about an order of magnitude for the various cold-worked states developed in this investigation.

DISCUSSION

Although it is somewhat premature to attempt a complete analysis of the data reported here at this time, some interesting deductions might nevertheless be made. The data for cold-worked and partially recovered aluminum are consistent with the previously reported data in that the high temperature creep rate of metals is given by

$$\dot{\varepsilon} = S e^{-\Delta H_c/RT} e^{B\sigma} \quad . \qquad (4)$$

These results demand that the usual models for creep, consisting either of thermal activation of dislocations over barriers[13] or local fluctuations of strain energy[14], be discarded since they all require that the stress parameter B be proportional to the reciprocal of the absolute temperature. An alternate model for creep has been suggested[15] which is based on the hypothesis that the rate-controlling process consists of the recovery of barriers and that dislocations migrate to the next barrier whenever the barrier strengths recover to the value of the applied stress. Since the barriers themselves are often considered as dislocations or patterns of dislocations, the unit recovery process can be self-diffusion, consisting of vacancy-atom exchanges at the center of the dislocations.

According to the usual models for creep[13,14], cold work would be expected to increase the barrier heights and thus the activation energy for creep. This prediction is contrary to the reported observation that ΔH_c is independent of the initial state of the metal. On the basis of the recovery model, however, the ΔH_c for the creep of an initially cold-worked metal should remain equal to that for self-diffusion in complete harmony with the facts reported here.

On the basis of the model of thermal activation of dislocations over a barrier, B is proportional to the so-called volume of a dislocation divided by the absolute temperature. It is possible that an entire dislocation could not be activated at one time but that it loops over the barriers in segments. If the length of each loop were determined by the spacing of the barriers it is possible that cold work, which might introduce more closely spaced barriers, would result in the looping of smaller segments of the dislocation, causing B to be smaller than that for an annealed material. But the previously proven fact that B is independent of temperature[10] disqualifies the usual model for creep based on the thermal activation of dislocations over a potential barrier. On the other hand the recovery model for creep does not yet permit an unqualified conclusion as to why $1/B$ is greater for the cold-worked state. As is known, alloying increases $1/B$, apparently by increasing the barrier strengths. It would therefore be expected that vacancies, interstitials and dislocations introduced during cold work could well cause $1/B$ to increase in a manner analogous to the effect of solid solution alloying. But the high temperature recovery annealing treatment following cold work and preliminary to the application of the creep stress, should have restored the vacancy and interstitial patterns to their equilibrium states. Perhaps the high $1/B$ values for the cold-worked and partially recovered aluminum are attributable to the non-equilibrium retention of some vacancies and interstitials in low energy states in appropriate strain centers or to the greater interaction forces arising from an overall increase in the number of dislocations. The gradual approach of $1/B$ with creep from that of the cold-worked and partially recovered metal to that for an initially annealed metal suggests the rather gradual approach to a steady state structure. Even though the picture on how $1/B$ is increased by cold work is yet very incomplete the experimental results do not disqualify the recovery model for creep.

The effect of structural changes during creep on the S-parameter can be incorporated into both the activation-over-barrier models[13,14] and the recovery model for high temperature creep. On the basis of the more acceptable recovery model for creep, cold working should increase the density of barriers and also increase the number of dislocations present. Whereas the first factor would decrease S , the second would increase S . Since there is as yet no apriori justification for assuming which of these two factors might predominate, the probable increase in S following cold work and partial recovery as suggested by the results in Fig. 6 is not a critical observation. The increase in S due to cold working, however, does not appear to have nearly as important an effect on the creep rate as the exponential effect of an increase in $1/B$ due to cold working.

High values of ΔH_c and low values of B and S are conducive to high creep resistance at elevated temperatures. The principal effect of cold working on the creep resistance appears to be primarily attributable to the effect of cold work on decreasing B. Additional investigations, covering a wider range of cold-worked states, however, will have to be made in order to ascertain more completely the effect of cold work on S.

CONCLUSIONS

1. The high temperature creep of cold-worked as well as annealed aluminum can be represented by the equation

$$\dot{\varepsilon} = S e^{-\Delta H_c/RT} e^{B\sigma}$$

where $\dot{\varepsilon}$ = creep rate

ΔH_c = activation energy for creep

R = gas constant

T = absolute temperature

σ = applied stress

B = stress parameter

and S = structure parameter.

2. The activation energy for high temperature creep is unaffected by previous cold work. This fact, as well as others, disqualifies the commonly postulated model for creep that is based on the assumption that the rate-controlling process for creep consists of thermal activation of dislocations over barriers.

3. The value of $1/B$ for initially cold-worked and partially recovered aluminum is greater than that for aluminum initially in the annealed state. However, as creep continues $1/B$ appears to approach that for the annealed metal.

4. S appears to be slightly larger for the initially cold-worked and partially recovered state than for the annealed state when compared at the same total creep strain and the same creep stress.

5. The principal effect of cold work on the creep resistance of aluminum appears to arise from its effect on increasing $1/B$.

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